STANDARD OPERATING PROCEDURE

FOR FIELD ANALYSIS OF TOTAL POLYNUCLEAR AROMATIC HYDROCARBONS

1 INTRODUCTION

The UVF-3100 Analyzer (SiteLab) provides a field test for total polynuclear aromatic hydrocarbons (PAHs). This SOP describes the application of the UVF-3100 method to sediment samples.

2 MATERIALS

- UVF-3100 Analyzer, which includes analyzer, scale, spatulas, adjustable pipetter, solvent dispenser, tissues, markers, and software.
- Sample Extraction Kit provided by SiteLab, which includes test tubes, filters, extraction jars, syringes, and pipette tips.
- HPLC-grade methanol.

3 PROCEDURES

- With a digital scale and stainless steel spatula, weight 5 grams of soil into an extraction jar labeled with the sample ID. Weight must be within +/- 0.1 g of 5 g.
- Into a test tube provided with the UVF-3100, add methanol to the 10 mL line. Empty into the extraction jar to create a 2x extract dilution.
- Shake soil jars by hand for several minutes. Let soil extract jars settle for a few minutes (at least 1 minute after solution has visibly settled).
- Using a syringe, remove between 2 and 4 mL of extract from the jar's surface. Attach a disposable filter to the syringe and dispense contents into test tube. Label extract with sample ID and dilution factor.
- Prepare a second dilution for analysis in a test tube labeled with sample ID and dilution factor. Using pipette provided with the UVF-3100, attach a new tip and dispense appropriate volume of dilution solution into test tube, as shown in Table 1. Add solvent to the appropriate line, for dilution as shown in Table 1.
- Pour the dilute solution into the glass cuvette. Cuvette must be at least 3/4 full. Using tissue wipes, clean outside of glass to remove fingerprints and liquids. Slide cuvette into the black cuvette holder.
- Lower cuvette holder into analyze and close the lid. Cuvette holder's arrow shaped handle must point to the silver dot at the left of the chamber on the UVF 3100A.
- When readout has stabilized after a few second, note reading. Multiply by dilution factor to obtain initial value.

- If readout is not within the detection range of the instrument, adjust dilution according to Table 1 and repeat analysis.
- Between samples, cuvette should be cleaned by rinsing with solvent into a waste jar.

4 QUALITY CONTROL

4.1 Definitions

- **Initial Calibration:** Calibration with 5 solutions provided by manufacturer, run on instrument start-up to ensure accuracy of instrument. Calibration curve should be linear.
- **Continuing Calibration:** Verify of curve linearity and check for drift by testing one or two standards as if they were samples.
- **Blank:** Pure methanol solution used similarly to continuing calibration.
- **Field Duplicate:** Duplicate sample removed from sediment grab to measure homogeneity within grab.
- **Extract Duplicate:** Duplicate of sediment extract that evaluates precision of dilutions and analyzer.

4.2 Calibration Procedures

- Allow instrument to warm up for at least 5 minutes. Rotate filter cylinder so that Slot A emissions optics are aligned next to the silver dot. Press "ENT" then "2" and enter the proper Maximum Range setting, as specified on calibration kit certificate.
- Begin with highest concentration calibration solution. Pour calibrator into cuvette and lower into analyzer. Enter correct concentration. Pour calibration solution back into test tube when finished. Repeat with remaining calibration solutions.
- View curve and test results using software provided with UVF-3100.
- For blank calibration, fill cuvette with clean methanol, and analyze when prompted at end of calibration. Wait for value to stabilize before pressing zero.

5 HEALTH AND SAFETY PROCEDURES

Methanol is a very flammable liquid. Methanol should be dispensed into small bottles for use, used under adequate ventilation, and not be stored near heat, flames, or sparks. Proper disposal of waste product is required. For more information, refer to the attached MSDS for methanol. (We will receive an MSDS from manufacturer with the methanol.)

Table 1. Dilution Factors			
Initial Solution	**Volume**	**Solvent Make-up Volume**	**Resultion Dilution**
2x	100 μL	5 mL	100x
2x	40 μL	10 mL	500x
2x	20 μL	10 mL	1000x
100x	100 μL	10 mL	10000x

Table 2. Quality Assurance Criteria			
Parameter	**Frequency**	**Criteria**	**Corrective Action**
Initial Calibration	At beginning of project and as needed for corrective action	r^2 value of above 0.9	Re-run calibration; contact manufacturer
Continuing Calibration	On instrument start up, after every 20 field samples, and at end of each day.	+/- 25% of actual value	Rinse cuvette and repeat analysis of calibration sample; If acceptable, perform duplicate analysis on last field sample. If RPD > 25% for last field sample, re-run previous batch.
Blank	With Continuing Calibration	Below detection limit	Rinse cuvette and repeat measurement with new blank solution. Perform blank calibration if blank remains above detection limit.
Field Duplicate	With every 20 field samples	n/a	For evaluation purposes only
Extract Duplicate	With every 20 field samples	RPD < 25%	Verify that concentrations are within detection range. Reanalyze extracts for previous batch. If RPD consistently > 25%, re-run calibration. If problem persists, contact manufacturer.

Note: RPD is calculated as follows. R_1 = larger of two observed values; R_2 = smaller of two observed values

$$\frac{R1 - R2}{(R1 + R2)/2} \times 100 = \%RPD$$